

Inhibition effect of some aromatic amines on copper electrodeposition from acidic baths

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Abstract To improve the quality requirements for copper deposits, the influence of some inhibition agents added to the acidic copper bath has been studied. Several aromatic nitrogen compounds have been tested as inhibition agents, such as aniline, *N*-methylaniline, *N*-ethylaniline, *N,N*-dimethylaniline (DMA), and *N,N*-diethylaniline (DEA). The electrochemical behavior of these organic additives and the most relevant aspects of the electrochemical behavior of copper in acid solutions have been analyzed by cyclic voltammetry. At the same time, a correlation between voltammetric data and molecular properties of protonated amines obtained by molecular modeling has been performed. The morphology of the copper deposits obtained in the absence and presence of organic compounds has been studied by scanning electron microscopy. An improvement of the roughness degree and crystallite size upon addition of aromatic amine has been found. Best results were obtained for DMA and DEA working at room temperature (25 ± 0.1 °C) and 200 A m^{-2} current density, in 1 mol L^{-1} sulfuric acid solution with $50 \text{ g L}^{-1} \text{ Cu}^{2+}$.

Keywords Acid copper bath · Inhibition agents · Aromatic amines · Molecular modeling

1 Introduction

Copper electrodeposition from acidic electroplating baths is the most common method to obtain interconnectors for microelectronics. The composition of acidic electroplating bath is mainly based on copper sulfate, sulfuric acid, promoters, such as chloride anions, brightening and leveling agents [1, 2]. It is well known that compact, adherent, and microcrystalline deposits are obtained only in the presence of inhibition agents (brightening and leveling agents), which are usually organic substances adsorbed at the metal–electrolyte solution interface. These substances are able to modify the kinetics of Cu(II) reduction to metallic copper and/or the crystallization process of copper atoms [3]. The presence of such organic compounds in the electroplating bath results in the improvement of the deposit properties, such as luster, hardness, roughness, and ductility. Among the organic compounds used as inhibition agents in acidic copper plating bath, thiourea has been extensively studied. It has been shown that thiourea acts through a strong interaction between the unsaturated sulfur atom and copper atoms [4]. Under these circumstances, the drawback is the inclusion of thiourea molecules or fragments in the cathodic deposit. Other addition agents containing sulfur as heteroatom are bis(3-sulfopropyl) disulfide [5, 6] and 3-mercapto-1-propanesulfonate [7, 8]. The adsorption of inhibition agents can also occur if the heteroatom is another electronegative atom such as oxygen or nitrogen. Commonly used brighteners and leveling agents include polyethylene glycol, polypropylene glycol [2, 9], and coumarine [10], as well as benzotriazole [11], saccharin [1, 12, 13], and picolinic acid [14]. The argument to use organic-nitrogen compounds is that the interaction is at least as strong as in case of sulfur compounds due to high adsorption energy at the metallic surface [15]. The benefit

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of using inhibition agents with nitrogen or oxygen heteroatoms is that copper deposits can be obtained free of foreign inclusions that could decrease their electrical conductivity. It is worth mentioning that aromatic amines are scarcely studied as inhibition agents in acidic copper electrodeposition. The influence of benzylamine and combined influence of benzylamine and chloride ions on copper electrodeposition from acidic baths have been reported [16, 17].

To predict the effect of organic additives on copper electrodeposition it is important to bear in mind the most relevant aspects of electrochemical behavior of copper in acidic media. Concerning the anodic behavior of copper in sulfuric acid solution, in addition to the predominant reaction of metallic copper dissolution to form Cu(II) ions, other processes may interfere that lead to surface passivation. Some authors admit that Cu(I) and Cu(II) oxides formed by hydrolysis undergo further transformations leading to Cu(I) and Cu(II) sulfates [18]. Other authors suggest that anodic copper passivation takes place by the direct formation of a Cu(II) sulfate film [19–21]. Analyzing the passive layer on the surface of copper, obtained by anodic dissolution of copper in weak acid solutions, Moreira et al. [22] concluded that it consists of a mixture of copper oxides near the metal and copper(II) sulfate in the vicinity of the electrolyte solution. Unlike weak acid solutions, in strong acid solutions, such as those used in copper electroplating baths, the thermodynamically stable species is copper(II) sulfate. Regarding the cathodic behavior of copper, it consists basically of two processes: the reduction of Cu(II) to Cu(I) which is the rate determining step and the fast reduction of Cu(I) to metallic Cu. Both processes are extensively reported in the literature [23, 24].

This article aims to study the electrochemical behavior of some aromatic nitrogen derivatives to identify additives with a superior inhibition effect. The following aromatic amines have been chosen for this study: aniline (A), *N*-methylaniline (MA), *N*-ethylaniline (EA), *N,N*-dimethylaniline (DMA), and *N,N*-diethylaniline (DEA). Several aspects have been evaluated in the attempt to establish the inhibitory capabilities, i.e., electrochemical stability studied by cyclic voltammetry, magnitude of dipole moment obtained by molecular modeling, influence on the kinetic parameters (Tafel slope and exchange current density) of copper electrodeposition, influence on the morphology of copper deposits studied by scanning electron microscopy.

2 Experimental

2.1 Chemicals

Aniline (for synthesis, $\geq 99\%$ GC), *N*-methylaniline (for synthesis $\geq 98\%$ GC), *N*-ethylaniline (for synthesis $\geq 98\%$

GC) *N,N*-dimethylaniline (for synthesis $\geq 99\%$ GC), and *N,N*-diethylaniline (for synthesis $\geq 99\%$ GC) were purchased from Merck and used as received. Sulfuric acid (Merck, p.a., 95–97%) and copper sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, Merck, $\geq 98\%$) were used to prepare the aqueous electrolyte solutions.

2.2 Electrochemical measurements

Linear and cyclic voltammograms were recorded using a Princeton Applied Research (PAR) 2273 potentiostat/galvanostat, from $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solutions containing $10 \text{ g L}^{-1} \text{ Cu(II)}$ and 0.5 mL L^{-1} aromatic amine. A conventional three-electrode one-compartment glass cell was used for the electrochemical measurements. The working electrode was a copper rod encapsulated in a plastic material holder so that the exposed surface area in contact with the electrolyte solution was 0.5 cm^2 . To evaluate the electrochemical stability of aromatic amines a platinum working electrode was used with a surface area of 0.07 cm^2 . To assure a reproducible surface, the copper working electrode was subjected to metallographic preparation with Struers LapoPol-21 by grinding and polishing using different grades emery paper and diamond spray up to $1 \mu\text{m}$, followed by cleaning in ultrasonic bath with distilled water. The counter electrode was a platinum wire and as reference a saturated silver–silver chloride electrode was used. All measurements were performed at $25 \pm 0.1^\circ \text{C}$ in stationary solutions. Dissolved oxygen was removed by purging solutions with high purity nitrogen before each measurement and a nitrogen blanket was kept above the solution during measurement.

2.3 Surface morphology

The surface morphology of copper deposits obtained in the presence of the studied aromatic amines was investigated by scanning electron microscopy using a FEI INSPECT S microscope.

3 Results and discussion

3.1 Some relevant aspects of electrochemical behavior of copper in sulfuric acid solution

As the reproducibility of the cyclic voltammograms on copper strongly depends on the working conditions and it is difficult to compare results reported in different articles, it was necessary to record the voltammograms in the real working conditions used in this study, to evaluate the electrochemical behavior of copper and to be able to predict the inhibition effect of selected organic additives.

Figure 1 shows a typical cyclic voltammogram recorded on copper in $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solution at a scan rate of 25 mV s^{-1} , in the potential range -0.7 to $+2.0 \text{ V}$. The potential range was chosen so that information could be obtained on both cathodic and anodic behavior, as during copper electrodeposition the addition agents are in contact with both the anode and cathode of the electroplating bath.

By sweeping the potential from the open circuit potential to more positive values, a pronounced increase in current is observed, due to the ionization of metallic copper to form copper ions. The accumulation of Cu^{2+} ions at the metal–electrolyte interface leads to copper passivation. The critical current density of about 0.5 A cm^{-2} is reached at an electrode potential around $+1.0 \text{ V}$ (peak A). It can be observed from Fig. 1a that after the passive state is reached, the current density becomes stable at about

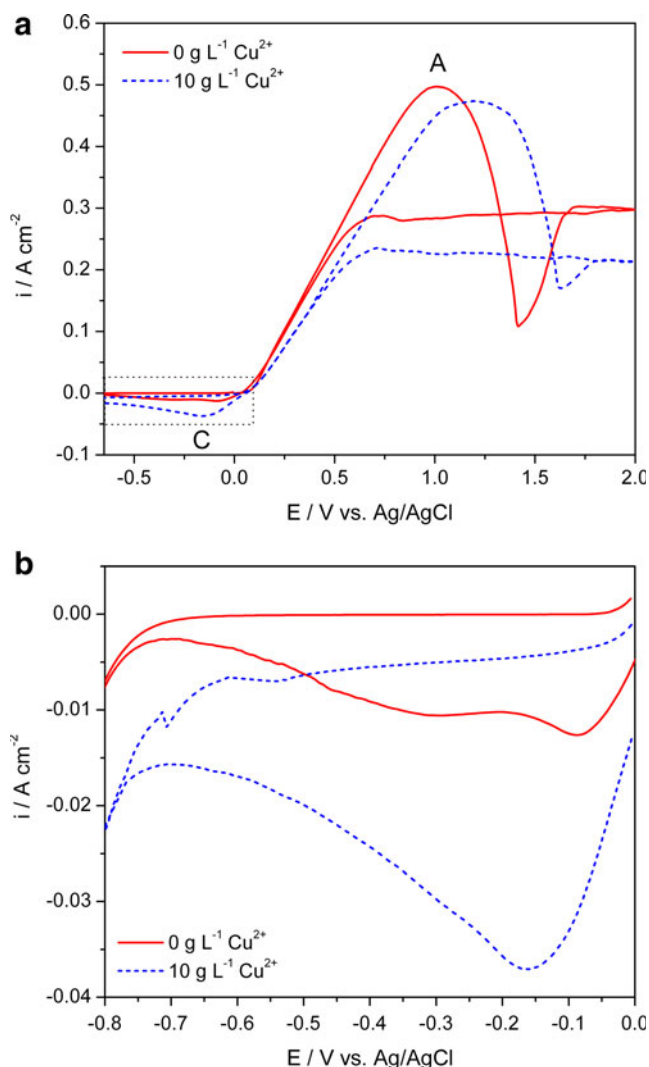


Fig. 1 Cyclic voltammograms on copper in $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solution with and without Cu^{2+} ions (a); enlargement of the cathodic branch (b). Scan rate 25 mV s^{-1}

0.3 A cm^{-2} , corresponding to the superficial film thickening and recovery.

The presence of Cu^{2+} ions in the electrolyte solution leads to a decrease of the critical current density necessary for passivation, which strengthens the idea of a passive layer based on Cu(II) compounds. By cathodic polarization, a small reduction peak appears in the solution containing Cu(II) ions, attributed to the reduction of Cu(II) ions present in the solution. Otherwise, in the solution without Cu(II) ions, the reduction peak is insignificant (Fig. 1b). At more negative potentials, below -0.55 V the hydrogen evolution reaction starts. A similar shape of the cyclic voltammograms was reported by Shinde et al. [25], who studied the electrochemical behavior of copper in oxalic acid solution and found that the passive layer on the metal surface consists of copper oxalate.

As expected, the critical current density for passivation depends on the scan rate, as the electrical charge across the interface is inversely proportional to the scan rate. In $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solution the critical current density is 0.65 A cm^{-2} at 50 mV s^{-1} , 0.75 A cm^{-2} at 125 mV s^{-1} and increases to a large value around 1.15 A cm^{-2} at higher scan rate 200 mV s^{-1} , as it can be observed in Fig. 2.

An interesting feature is the appearance on the reverse run of the cyclic voltammograms of a less pronounced but completely reproducible oxidation peak A^* . It can be explained by the dissolution of the passive layer when the anodic polarization is lowered. Thus, metallic copper is again in contact with the electrolyte solution and because the potential is still more positive than the equilibrium potential it can be oxidized as shown by the slight increase

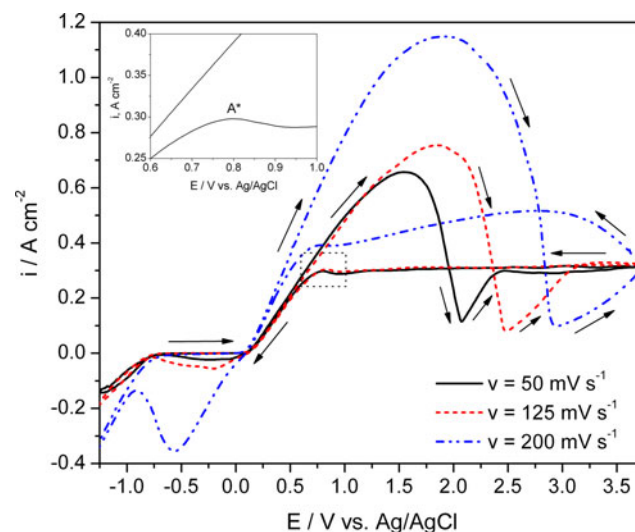


Fig. 2 Cyclic voltammograms on copper in $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solution at different scan rates (50 , 125 , and 200 mV s^{-1}). The inset shows an enlargement of the oxidation peak A^* for 50 mV s^{-1}

in current. Thereafter, the current starts to decrease rapidly as the potential comes closer to the equilibrium value.

3.2 Voltammetric study of electrochemical behavior of aromatic amines

In addition to a benefic influence on the morphology of metallic deposits, addition agents should fulfill another fundamental condition, they must be electrochemically inactive. For this reason, it is necessary to investigate the electrochemical behavior of addition agents over the entire accessible potential range in the solution, as they accede both to the anode and cathode.

To avoid interferences of the electrochemical reactions of copper and copper ions, the electrochemical behavior of aromatic amines was studied on platinum electrode in 1 mol L⁻¹ sulfuric acid solution, without the addition of copper ions. Figure 3a, b show cyclic voltammograms on platinum in the absence and presence of 0.5 mL L⁻¹ added

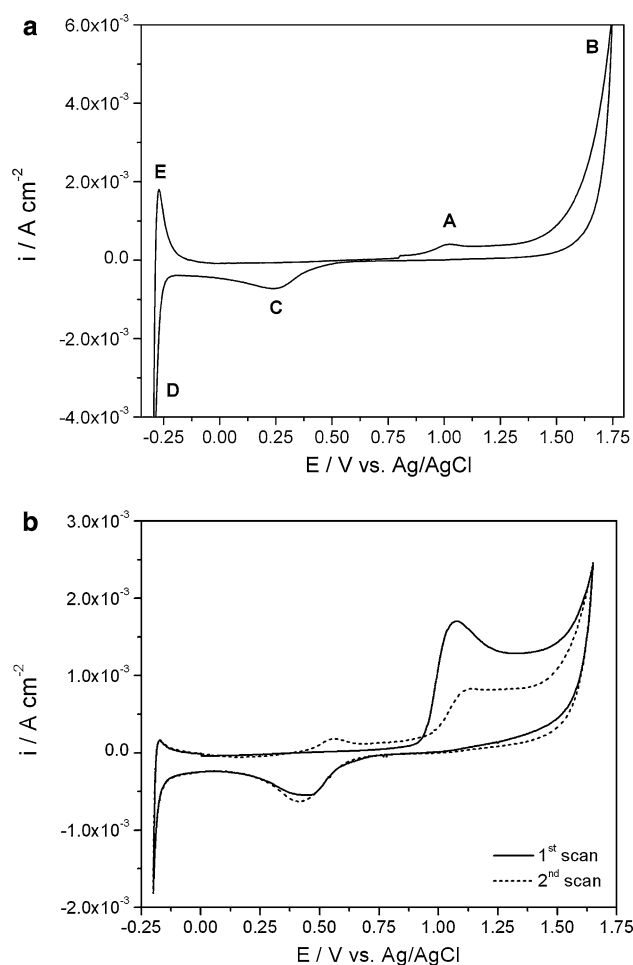


Fig. 3 Cyclic voltammograms on Pt in 1 mol L⁻¹ H₂SO₄ solution in the absence (a) and presence of 0.5 mL L⁻¹ A (b). Scan rate 2.5 mV s⁻¹

aniline. The electrochemical behavior of Pt in sulfuric acid solution is well known [26] and is characterized by several peaks corresponding to the oxidation of hydrogen adsorbed on the surface of the electrode (peak E), followed by the formation of superficial oxides (peak A) which are reduced on the reverse run (peak C). At high positive potentials the water oxidation takes place with evolution of oxygen (curve B) and at negative potentials the reduction of protons and hydrogen evolution can be observed (curve D).

In the presence of aniline a new anodic peak appears at 1.07 V, corresponding to the oxidation of protonated aniline molecule to phenylammonium radical cation. The radical cation initiates the polymerization process and reacts with a monomer molecule to form a dimer. The mechanism of aniline polymerization is extensively described in the literature [27–29] by the formation of radical cations which initiate the polymerization process by reacting with monomer molecules and forming dimers. Dimers react further to form oligomers and finally longer polymer chains. For polyaniline three idealized oxidation states are possible: leucoemeraldine, the fully reduced form, emeraldine, the partially oxidized form and pernigraniline, the completely oxidized form. On the reverse run of the cyclic voltammogram a reduction peak of the oxidation products appears at 0.41 V. In the second cycle, a new oxidation peak at 0.56 V corresponds to degradation products such as *p*-benzoquinone [30] formed by overoxidation reactions due to the high anodic limit used in the cyclic voltammograms.

A similar behavior was observed for all the studied aromatic amines except for the oxidation peak potential. The substitution of the hydrogen atoms of the amino group by one or two alkyl radicals changes the oxidation onset potential of the monomers, as it can be seen in Fig. 4.

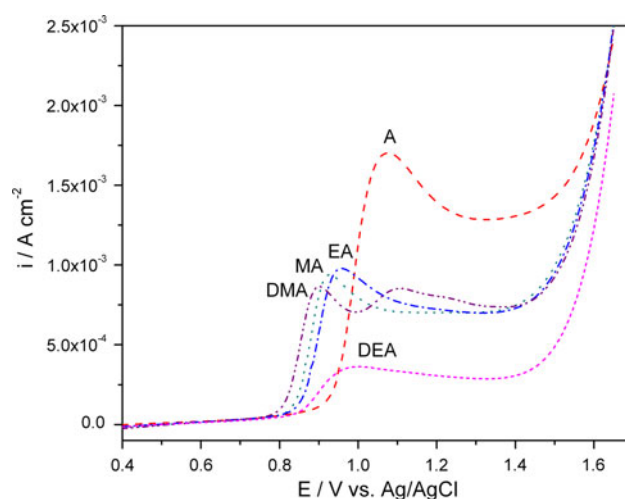


Fig. 4 Anodic branch of cyclic voltammograms on Pt in 1 mol L⁻¹ H₂SO₄ solution in the presence of 0.5 mL L⁻¹ added aromatic amines. Scan rate 2.5 mV s⁻¹

Alkyl groups have an electron-releasing inductive effect, the more branched the alkyl groups are, the more important is the inductive effect. As a result, the oxidation potential of *N*-alkylanilines will decrease compared to that of aniline. Accordingly, the onset oxidation potential decreases in the order: A (0.94 V) < MA (0.84 V) < DMA (0.81 V). However, the polymer film formation in case of *N*-alkylanilines is obstructed by two reasons. On the one hand, the electrophilic attack on the nitrogen atom is sterically hindered by the voluminous substituents and on the other hand, the insertion of side groups increases the solubility of the oligomeric products [31]. Studies on the electropolymerization mechanism of MA in aqueous solutions and aqueous-organic solvent mixtures have shown large amounts of soluble reaction products [32–34].

The onset potential of the oxidation of monomers is much more positive (over +0.8 V) than the copper oxidation potential in highly acidic media ($E < +0.45$ V vs. NHE [22]) allowing us to conclude that, on the copper anodes used in copper electroplating, the addition agents will not suffer any oxidation processes. In addition, the relatively low current density values applied in copper electroplating are not large enough to allow an advanced polarization of the anode and to install the passive state.

3.3 The influence of aromatic amine on copper electrodeposition from acidic solutions

3.3.1 Molecular modeling of the addition agents

Supplemental information about amines behavior at copper–electrolyte solution interface can be obtained by molecular modeling. Ab-initio computations were performed using GAMESS-US program [35] to optimize the geometry of molecules and to compute dipole moments at 6-31G(d,p) level. Other molecular parameters (molecular van der Waals volume and covered area normalized on the dipole moment) were also computed [36] and the obtained values are given in Table 1.

The protonated anilines dipole moment decreases from aniline to DEA, according to the donor inductive effect of alkyl substituents. Higher dipole moment values of protonated amines ensure a normalized orientation of dipole moment on the copper surface due to the electric field at

metal–electrolyte solution interface. Therefore, one can conclude that the main factor influencing the shielding capacity of metal is the greatest molecular area section normalized to the dipole moment direction. Obviously, one have to emphasize that there is a steric hindrance of ions access to the metal surface due to the molecular van der Waals volume of the protonated amines. The hydrophobic character of alkyl substituents is another limiting parameter for the ions access to metal surface.

3.3.2 Kinetics of copper electrodeposition in the presence of addition agents

To identify the aromatic amines with the most pronounced inhibition effect in acidic copper plating baths, their influence on the cathodic copper deposition process was studied by linear sweep voltammetry. Figure 5 shows linear voltammograms obtained on copper electrode, during copper electrodeposition in the absence and presence of organic additives.

As it can be observed in Fig. 5, the cathodic polarization of the copper electrode in a solution containing Cu^{2+} ions and the studied aromatic amines resulted in all cases in a shift of the reduction peak potential to more negative values in conjunction with a decrease of the peak current and also of the limiting current density. The changes in the linear voltammograms induced by the presence of these organic additives can be explained in terms of adsorption of ammonium cations on the copper electrode surface. Moreover, there is a clear dependence between overpotential shift and decrease of the limiting current density on one hand and the molecular properties of amines on the

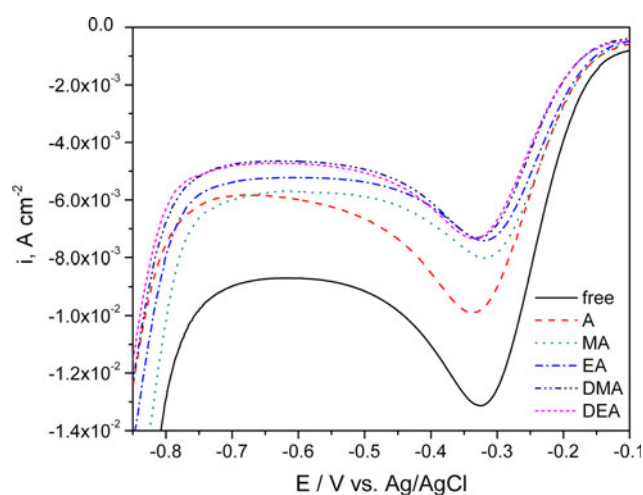


Fig. 5 Linear voltammograms on Cu electrode from acid solutions ($10 \text{ g L}^{-1} \text{ Cu}^{2+}$; $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$) in the absence and presence of amines. Scan rate 10 mV s^{-1} . Electrode potentials are measured versus the open circuit potential (OPC)

Table 1 Computed molecular parameters for selected anilines

Molecule	Volume [\AA^3]	Surface [\AA^2]	Dipole moment [D]
$[\text{AH}]^+$	97.2	12.0	7.5
$[\text{MAH}]^+$	113.5	14.1	6.3
$[\text{EAH}]^+$	131.3	17.1	4.7
$[\text{DMAH}]^+$	129.6	14.6	5.5
$[\text{DEAH}]^+$	161.3	20.5	3.2

other hand. The least influence is observed in case of A, as its volume and surface coverage are the lowest. For MA and EA the influence is moderate, whereas for DMA and DEA the most pronounced effect is obtained regarding the decrease of limiting current density. It is assumed that the presence of two hydrophobic substituents (methyl and ethyl) further limits the access of hydrated Cu^{2+} ions to the cathode.

The kinetic parameters for copper electrodeposition (charge transfer coefficient α_c and exchange current density i_0) have been determined from the linear part of the Tafel plots, in a potential range where the rate determining step is the charge transfer process. The linear Tafel plots, corrected for the ohmic drop in the solution, are given in Fig. 6.

The calculated values of the kinetic parameters are given in Table 2. According to the obtained results, it can be observed that the Tafel slope decreases moderately in the presence of any of the studied amines, from 120 mV decade⁻¹ in an additive-free solution to 114 mV decade⁻¹ (the lowest value) in the presence of DEA. In accordance with the Tafel slope decrease there is a slight increase in the charge transfer coefficient α_c from 0.49 in the absence of additives to 0.52 in the presence of DEA, indicating that the presence of aromatic amines does not change the mechanism of cathodic copper deposition.

A more significant influence of the aromatic amines is observed on the values of the exchange current density. The higher the volume and surface coverage of amines molecules, the lower is the exchange current density. It can be concluded that the inhibition effect on the copper deposition process is due to the adsorption on the cathode surface. The values obtained for the Tafel slope b , cathodic

Table 2 Tafel fitting results for copper deposition

Organic additive	$-b$ [mV dec ⁻¹]	α_c	$i_0 \times 10^4$ [A cm ⁻²]
–	120	0.49	2.64
A	119	0.50	1.97
MA	118	0.50	2.07
EA	116	0.51	2.01
DMA	115	0.51	1.51
DEA	114	0.52	1.22

charge transfer coefficient α_c and exchange current density i_0 are in good agreement with literature data [37–39].

3.3.3 Morphological characterization of the cathodic deposit

The influence of aromatic amines on the morphology of copper deposits was studied on samples obtained at a current density of 200 A m⁻² for 10 min, from 1 mol L⁻¹ H₂SO₄ solution with 50 g L⁻¹ Cu²⁺ and 0.5 mL L⁻¹ organic additives. Figure 7a–f show SEM micrographs of the cathodic copper deposit obtained in the absence and presence of the studied addition agents.

It can be observed in Fig. 7a that without any additive the copper deposit appears as agglomerated metallic particles, macrocrystalline, and rough. Generally, the addition of aromatic amines as inhibition agents induces a reorientation of the metallic particles and a decrease of their sizes. The copper deposits obtained in the presence of A and MA show an improvement of the roughness degree, still they are not completely satisfactory. The best results in terms of roughness degree and crystallite size were obtained for DMA and DEA for the specific electrolyte solution composition and current density value used in this study.

4 Conclusions

To investigate the addition agent properties of the selected aromatic nitrogen compounds, cyclic voltammetry has been used. As the reproducibility of polarization curves on solid electrodes is rather unsatisfactory, it was necessary to study the electrochemical behavior of copper in the particular conditions used to test the addition agents. The cyclic polarization curves on copper in sulfuric acid, with or without Cu²⁺ ions, revealed the most relevant aspects of electrochemical behavior of copper: the presence of passive state at anodic polarization, the dissolution of the passive film by sweeping the potential to more negative values and the partial reduction of Cu²⁺ ions originating from the passive film dissolution at cathodic potentials. An interesting detail of such polarization curves is the removal

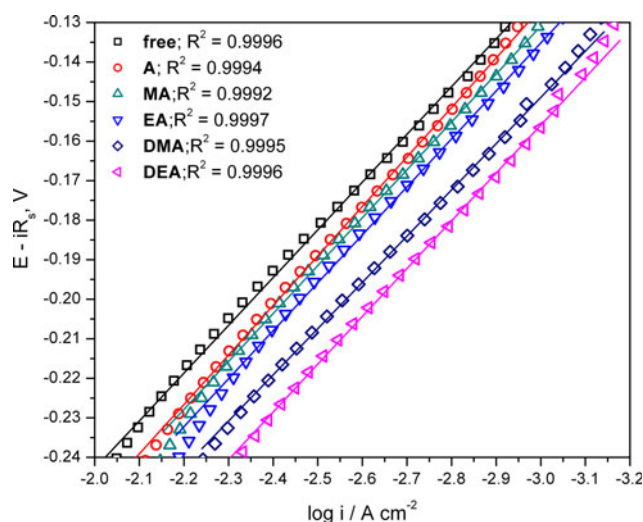


Fig. 6 Tafel plots for copper electrodeposition in 1 mol L⁻¹ H₂SO₄ solution with 10 g L⁻¹ Cu²⁺, in the absence and presence of aromatic amines

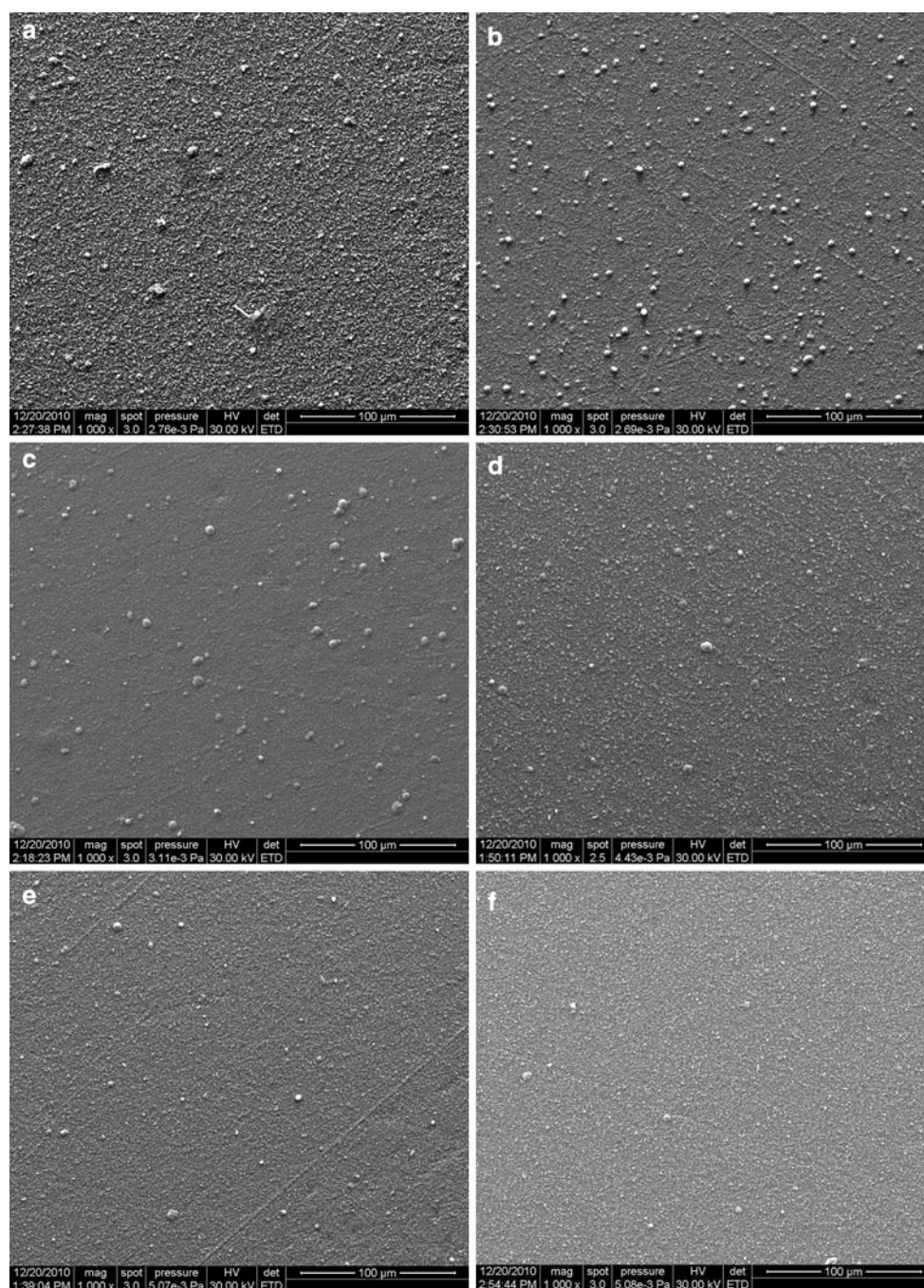


Fig. 7 SEM micrographs of copper deposits obtained at 200 A m^{-2} from $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solution with $50 \text{ g L}^{-1} \text{ Cu}^{2+}$ without additives (a) and with additives: A (b), MA (c), DMA (d), EA (e), and DEA (f). Magnitude $1000\times$

of the passive film by a merely physical process, when the potential is swept in cathodic direction but before the reversible potential is reached.

Cyclic voltammetry was also used to demonstrate that the studied amines do not undergo electrochemical transformations in the potential range accessible in a copper electroplating bath. This is an essential aspect, as the addition agents in a plating bath come in contact with both

anodes and cathodes. On the other hand, the current densities applied in industrial copper electroplating are rather reduced and not sufficient to allow an advanced polarization at which aromatic amines start to become electroactive.

Molecular modeling of addition agents brought supplementary information about the mechanism of action on the copper-electrolyte surface. It should be mentioned that in

such high acidic solutions the aromatic amines exist as phenylammonium ions. Due to a large dipole moment, the phenylammonium ions show a net orientation on the interface, perpendicularly to the cathode surface. For this reason, we have admitted that the shielding capacity of protonated amine molecules is equal to the maximum molecule section normal to the dipole moment.

Analyzing the parameters which characterize the kinetics of copper electrodeposition, it has been found that the effect of aromatic amines is significant on the exchange current density i_0 and less important on the cathodic charge transfer coefficient α_c . The fact that the effect of amines on the exchange current density is even more pronounced as their volume and surface coverage are higher, suggests that the inhibition effect on the copper electrodeposition process is a result of adsorption of phenylammonium ions at the cathode surface.

SEM micrographs of cathodic copper deposits obtained at 200 A m⁻² current density for 10 min, from a solution containing 50 g L⁻¹ Cu²⁺ ions, confirmed the previous conclusions and indicated that the best addition agent are DMA and DEA.

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